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THE EFFECT OF HYDROCARBON FILL FLUIDS ON 5109S NEOPRENE AND QXA 3770 FLUOROCARBON RUBBERS

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QXA 3770 FLUOROCARBON RUBBERS

John A. Hiltz — Irvin A. Keough

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Abstract

The chemical resistance of two elastomers, 5109S neoprene rubber and QXA 3770 fluorocarbon rubber, to three hydrocarbon-based buoyancy fluids has been assessed by monitoring weight gain and dimensional changes of rubber samples immersed in the fluids. The neoprene rubber absorbed approximately 7%, 20% and 20% by weight Bayol 35, Voltesso 35 and Marcol 72 respectively. The dimensions of the neoprene rubber also increased following immersion in the fluids. The fluorocarbon elastomer absorbed less than 0.15% by weight of the fill fluids, and did not swell following immersion in the fluids. Dynamic mechanical analysis was also used to monitor changes in the properties of the elastomers following immersion. The glass transition temperature (Tg) of the neoprene rubber samples decreased by 15 to 20 degrees following immersion in the fill fluids. Changes is the Tg of the fluorocarbon rubber following immersion were considerably smaller than those observed for the neoprene rubber. The results indicate that the fluorocarbon rubber has good chemical resistance to the fill fluids while the neoprene rubber does not. However, the dynamic properties of the fluorocarbon rubber indicate that it should not be used for low temperature, high frequency applications if rubber-like properties are required.

Résumé

On a évalué la résistance chimique de deux élastomères, soit le néoprène 5109S et le caoutchouc fluorocarboné QXA 3770, à trois liquides de flottabilité à base d'hydrocarbure, en déterminant le gain de poids et les variations dimensionnelles d'échantillons de caoutchouc plongés dans ces liquides. Le néoprène absorbait environ 7 %, 20 % et 20 % en poids de Bayol 35, de Voltesso 35 et de Marcol 72, respectivement. De plus, les dimensions du néoprène augmentaient après immersion dans les liquides. L'élastomère fluorocarboné absorbait moins de 0, 15% en poids de liquide et ne gonflait pas après immersion dans les liquides. L'analyse mécanique dynamique a aussi permis de déterminer les changements de propriété des élastomères après immersion. La température de transition vitreuse (T_g) des échantillons de néoprène diminuait de 15 à 20 degrés après immersion dans les liquides. Les variations de T_g du caoutchouc fluorocarboné étaient, après immersion, considérablement plus faibles que celles observées avec le néoprène. Selon les résultats, le caoutchouc fluorocarboné possède, contrairement au néoprène, une bonne résistance chimique aux liquides. Toutefois, les propriétés dynamiques du caoutchouc fluorocarboné indiquent que ce caoutchouc ne devrait pas être utilisé dans des applications très fréquentes à faible température, lorsque des propriétés caoutchouteuses sont exigées.

Executive Summary

DREA TM/98/203

The Effect of Hydrocarbon Fill Fluids on 5109S Neoprene and QXA 3770 Fluorocarbon
Rubbers
by
John A. Hiltz and Irvin A. Keough

The chemical resistance of two elastomers, 5109S neoprene rubber and QXA 3770 fluorocarbon rubber, to three hydrocarbon-based buoyancy fluids, has been assessed by monitoring weight gain, dimensional changes, and changes in the dynamic properties of rubber samples immersed in the fluids. One of the elastomers, 5109S neoprene, had been used as a boot material on barrel stave projectors and had swollen and failed while in service. In-service conditions included exposure to a hydrocarbon fill (buoyancy) fluid. The other elastomer, QXA 3770, was recommended for testing because of its reported excellent resistance to hydrocarbon fluids. The three fill fluids used were Bayol 35, Voltesso 35, and Marcol 72.

The neoprene rubber absorbed approximately 7%, 20% and 20% by weight Bayol 35, Voltesso 35 and Marcol 72 respectively. The dimensions of the neoprene rubber also increased following immersion in the fluids. The fluorocarbon elastomer absorbed less than 0.15% by weight of the fill fluids, and did not swell following immersion in the fluids. Dynamic mechanical thermal analysis was used to monitor changes in the dynamic properties of the elastomers following immersion. The glass transition temperature (Tg) of the neoprene rubber samples decreased by 15 to 20 degrees following immersion in the fill fluids. The effect of the fill fluids on the Tg of the fluorocarbon was considerably smaller.

The results indicate that the fluorocarbon rubber has good chemical resistance to the fill fluids while the neoprene rubber does not. Multiple fixed frequency testing and time temperature superpositioning analysis were used to determine the frequency dependence of the dynamic properties of rubber. The results indicated that QXA 3770 fluorocarbon was not suitable for use in applications where low temperature and frequencies greater than approximately 500 Hz were encountered.

The report recommends that epichlorohydrin rubber be considered for use as a boot material. Testing of the chemical resistance, processibility, and dynamic properties of epichlorohydrin rubber samples is in progress.

Table of Contents

1.0 Introduction

In applications where dimensional stability, load bearing properties, and the glass transition temperature (T_g) of a polymeric material are important, changes induced by absorbed fluid or loss of plasticizer can be critical. Absorption of a fluid can result in swelling, a reduction in tensile strength and modulus, an increase in elongation at break, and a decrease in the T_g of a polymer. The fluid may also leach additives, such as plasticizers, from a polymer. The result is often an opposite effect, i.e., loss of a plasticizer will lead to an increase in tensile strength and modulus, a decrease in elongation at break, and an increase in T_g of the polymer.

The effect of a fluid on the properties of a polymeric material can be assessed in a number of ways. One of these involves immersing the polymer in a fluid and monitoring absorption of the fluid (weight gain) with time. The rate of diffusion of the fluid into the polymer and weight of fluid absorbed by the elastomer give an indication of the resistance of the elastomer to the fluid. An absorbed fluid also effects the dynamic mechanical response of the elastomer. The magnitudes of the storage modulus (E'), loss modulus (E"), and tan δ (E"/E') of an elastomer are all effected by the absorption of a fluid. Similarly, loss of an additive, such as a plasticizer will also affect the dynamic mechanical response of an elastomer. Changes in the magnitude and temperature of the maximum value E" and tan d can be correlated with fluid absorption or plasticizer loss.

DREA was requested to evaluate the chemical resistance of a neoprene rubber, used as boot material on a barrel stave projector, to three hydrocarbon-based fill (buoyancy) fluids. It was reported that the neoprene rubber (5109S), when in contact with hydrocarbon-based fluids, would swell. This led to the pinching and cutting of the rubber between the barrel staves during operation of the projector. DREA was also asked to recommend and test an elastomer with excellent chemical resistance to hydrocarbon-based fluids. The fluorocarbon rubber selected was Fluorel QXA 3770.

1

In this memorandum, absorption (weight gain) versus immersion time data, dimensional changes, and changes in the glass transition temperature of the rubbers are used to assess the chemical resistance of the two elastomers to the fill fluids. Additionally, the frequency dependence of the E' and tan d of the fluorocarbon rubber is investigated to determine if this elastomer will remain rubber-like at lower temperatures (~0°C) and frequencies above 100Hz.

2.0 Experimental

2.1 Fluids

Three hydrocarbon fluids, Bayol 35, Voltesso 35, and Marcol 72, manufactured by Imperial Oil, Toronto, Ontario were used in the chemical resistance testing of the elastomers. These fluids are used in towed array applications where neutral buoyancy is required. The viscosity of these fluids also facilitates their use. They are easy to pour and problems arising from entrapped air are minimized.

Bayol 35 is described[1] as a mineral oil consisting of a mixture of paraffinic and/or naphthenic hydrocarbons. Paraffinic refers to straight chain saturated hydrocarbons, while naphthenic refers to cyclic saturated hydrocarbons. It is reported to have a viscosity of 3.42 centistokes (cSt) at 25°C, a boiling range from 205°C to 255°C, and a density of 0.79 g/cc at 15°C.

Voltesso 35 is described[2] as a lubricating oil consisting of a mixture of saturated and unsaturated hydrocarbons derived from naphthenic distillate and distillates. It is reported to have a viscosity of 8.00 cSt at 40°C, a boiling range from 229°C to 444°C, a freezing pour point of -51°C, and a density of 0.87 g/cc at 15°C.

Marcol 72 is described[3] as a white mineral oil that consists of a mixture of naphthenic hydrocarbons. It is reported to have a viscosity of 12.40 cSt at 40°C, a boiling point of 230°C, a freezing pour point of -9°C, and a density of 0.84 g/cc at 15°C.

2.2 Elastomers

5109S boot rubber is a neoprene rubber developed by the U. S. Navy for underwater applications. Its composition is listed in Table 1.

Table 1

Components and their relative concentrations in 5109S neoprene rubber formulation.

Component	Parts by weight
Neoprene GRT	100
Stearic acid	1
Octylated Diphenylamine	2
Benzothiazyl Disulfide	1.5
Red-Lead Dispersion(90% in EPDM)	15
TE-70 Processing Aid	2
N550 Carbon Black	31

Fluorel QXA 3770, is a 50/50 mixture of two 3M fluorocarbon elastomers, Fluorel FE-5642 and Fluorel FC-2174. The elastomer contains 65.9% fluorine by weight and is a copolymer of vinylidene fluoride and hexafluoropropylene.

2.3 Fluid Exposure

Samples of 5109S rubber (10cm X 6cm, 6g) and Fluorel QXA 3770 (8.5 cm X 4.5 cm, 13g) were immersed in Bayol 35, Voltesso 35, and Marcol 72 and weight gain and changes in length and width monitored with time. Excess fluid on the surface of the samples was wiped off prior to weighing.

2.4 Dynamic Mechanical Thermal Analysis (DMTA)

All DMTA was carried out on a DuPont Instruments Model 983 Dynamic Mechanical Analyzer with a liquid nitrogen cooling accessory. The analysis was done in the resonance mode using a temperature ramp of 5°C/min from -100°C to 50°C. A typical test sample was 45 mm long X 10 mm wide. Thickness of the test samples varied. The glass transition temperature (T_g) of the samples was taken as the maximum in the plot of loss modulus (E") versus temperature.

2.5 Gas Chromatography/Mass Spectrometry (GC/MS)

All GC/MS analyses were made on a Fisons Model 8000 capillary gas chromatograph quadrupole mass spectrometer in the full scan mode (25 atomic mass units (amu) to 500 amu) using a 30 m long X 0.25 mm inside diameter 100% methyl silicone column. The GC oven was programmed to hold at 40°C for 5 minutes, ramped at a rate of 10°C/min to 300°C, and then held at 300°C for 9 minutes.

3.0 Results and Discussion

3.1 Fluid Absorption

Plots of the percentage weight gain of 5109S neoprene rubber in Bayol 35, Voltesso 35 and Marcol 72 fill fluids against immersion time$^{1/2}$ are shown in Figure 1 respectively. The equilibrium percentage weight gain of 5109S was approximately 6.5 % in Bayol 35 and approximately 20% in both Voltesso 35 and Marcol 72. For all three fluids, the % weight gain reached a maximum and then decreased with continued exposure. This may be indicative of diffusion of additives in the rubber into the fill fluids.

Plots of percentage weight gain of Fluorel QXA 3770 in Bayol 35, Voltesso 35, and Marcol 72 fill fluids against immersion time$^{1/2}$ are show in Figure 2. The percentage weight gain of Fluorel QXA 3770 was greatest in Marcol 72, and was less than 0.15% in each of the three fluids. The plots indicate that equilibrium fluid concentrations in Fluorel QXA 3770 had not been reached after 2000 hours exposure.

The weight of hydrocarbon fluids, such as Bayol 35, Voltesso 35, and Marcol 72, absorbed by a polymeric material is dependent on a number of factors. These include the molecular weight of the hydrocarbons that make up the fluid, the structure of the hydrocarbons, and the structure and composition of the polymer. For instance, in a series of straight chain hydrocarbons, the diffusion of the lower molecular weight hydrocarbons into the rubber will take place more rapidly than the higher molecular weight hydrocarbons. However, the solubility of the higher molecular weight compounds in the rubber is greater than the lower molecular weight compounds. Straight chain

4

hydrocarbons may be absorbed more slowly than aromatic hydrocarbons or cyclic aliphatic hydrocarbons with the same molecular weight[4,5,6].

The structure of the polymer will have an effect on the rate at which a fluid is absorbed by the polymer. The chemical resistance of neoprene (polychloroprene) rubbers to hydrocarbons fluids, although not excellent, is better than rubbers such as polyisoprene, butyl, and polybutadiene, that do not contain chlorine. The polar nature of the chlorine atom retards the absorption of non-polar fluids, such as hydrocarbons. The fluorocarbon rubber contains a significantly higher percentage of polar fluorine atoms and has excellent resistance to hydrocarbon fluids. This is evident in the difference in the weight of the fill fluids absorbed by the neoprene and fluorocarbon rubbers.

3.2 Effect of Fluid Absorption on Glass Transition Temperature

The weight gains and the T_gs of the elastomer samples, following exposure to the fill fluids, are summarized in Table 2. In a DMTA experiment, the storage modulus decreases rapidly in the area of the glass transition while the loss modulus and tan δ go through a maximum in the area of the glass transition. A plot of loss modulus (E") versus temperature for 5109S neoprene rubber prior to immersion in the fill fluids is shown in Figure 3. A similar plot for a sample of 5109S neoprene rubber following immersion in Bayol 35 is shown in Figure 4. The T_g was taken as the maximum in the plot of E" versus temperature. The T_g decreased from $-31.9^{\circ}C$ to $-47.2^{\circ}C$ following absorption of 7% Bayol 35 indicating that the absorbed fill fluid has plasticized the rubber. Immersion of 5109S neoprene rubber in Voltesso 35 and Marcol 72 also led to a decrease in the T_g, from $-31.9^{\circ}C$ to $-46.9^{\circ}C$ and $-50.0^{\circ}C$ respectively.

The decrease in T_g of the neoprene rubber following exposure to the three fill fluids did not correlate well with the weight percent fill fluid absorbed. That is, the decrease in T_g resulting from the absorption of 6.54% Bayol 35 was similar to that resulting from absorption of approximately 20 % of the other fluids. This can be attributed to a number of factors.

5

Table 2

Percent weight gain and glass transitions temperature of samples of 5109S neoprene and QXA 3770 Fluorocarbon rubber following exposure to Bayol 35, Voltesso 35, and Marcol 72 hydrocarbon fill fluids.

FLUID			RUBBER	
	5109S Neoprene		QXA 3770 Fluorocarbon	
	% weight gain	T_g	% weight gain	T_g
none	0.00	-31.9	0.00	-11.2
Bayol 35	6.54	-47.2	0.09	-6.8
Voltesso 35	20.59	-46.9	0.06	-13.2
Marcol 72	19.82	-50.0	0.14	-7.9

The components of the fill fluids vary both in molecular weight and structure and these factors influence the effect a given weight of the fluids will have on the dynamic mechanical response of the polymer. In addition, leaching of additives from the elastomer into the fluids cannot be ruled out. The concurrent loss of an additive, such as a plasticizer, would increase the T_g of the polymer.

The T_g of the fluorocarbon rubber following exposure to the three fill fluids did not correlate with weight percent absorbed fluid either. Further, the T_g of the elastomer following exposure to Bayol 35 and Marcol 72 increased. This strongly suggests that something is being leached (extracted) from the polymer into the fill fluid.

To demonstrate that the increase in the T_g of the fluorocarbon rubber following exposure to the Bayol 35 fill fluid was due to loss of plasticizer, a sample of fluorocarbon rubber was immersed in Bayol 35 fill fluid. Gas chromatography/mass spectrometric (GC/MS) analysis of samples of Bayol 35 before and after 48 hours exposure to the fluorocarbon rubber are shown in Figure 5. A small peak can be seen at approximately 31 minutes in the chromatogram of the Bayol 35 sample that had been exposed to the fluorocarbon rubber. This area of the two chromatograms is expanded in Figure 6. The mass spectrum of the compound giving rise to this peak, along with 'best fit' as mass spectra matches,

6

are shown in Figure 7. The analysis indicates that the compound is diisodecylphthalate, a phthalic acid based plasticizer.

3.3 Dimensional Changes

As the boot material is used in an application where dimensional stability is important, changes in dimension of the materials were monitored during immersion testing.

The dimensions of samples of 5109S neoprene rubber changed from 10.0 cm X 4.8 cm to 10.6 cm X 5.1 cm following 400 hours immersion in Bayol 35, from 10.0 cm X 5.4 cm to 11.2 cm X 6.0 cm following 400 hours immersion in Voltesso 35, and from 10.0 cm X 5.1 cm to 10.9 cm X 5.6 cm following 400 hours immersion in Marcol 72. The dimensional changes in the 5109S neoprene rubber samples exposed to Bayol 35, Voltesso 35 and Marcol 72 correspond to 12.6%, 24.4% and 19.7% increases in surface area respectively. The percentage changes in surface area correlate loosely with the percentage weight gain of the rubber samples, i.e., the percentage increase in surface area and weight gains for Bayol 35 were 12.6% and 6.5%respectively, for Voltesso 35 were 24.4% and 20.6% respectively and for Marcol 72 were 19.7% and 19.8% respectively. Changes in the dimensions of Fluorel QXA 3770 fluorocarbon rubber in contact with the three fill fluids were negligible.

3.4 Dependence of T_g on Frequency

The dynamic response of polymeric materials is dependent on both temperature and frequency. That is, an increase in frequency will have the same effect on the dynamic properties as a decrease in temperature. This phenomenon is especially important when the service temperature of a polymer is close to the glass transition temperature. In this region, an increase in frequency can result in the response of the polymer changing from rubber like to glass like.

The T_g of QXA 3770 fluorocarbon rubber, measured in the resonance mode and (less than 50 Hz), was approximately -10°C. Service temperatures for this elastomer, that is, salt water immersion North Atlantic, may be less than 0°C. Therefore it is important to determine the dynamic response of the elastomer over an extended frequency range to ensure that it is still in the rubbery region in the working frequency range.

The time-temperature superposition principle[7] was used to determine the frequency response of this fluorocarbon elastomer over an extended frequency range. The dynamic properties (E' and tan δ against temperature) of QXA 3770 fluorocarbon rubber were measured at a number of frequencies and the data is shifted to a reference temperature (-5°C).

Figures 8 and 9 show plots of E' and tan d against temperature for QXA 3770 fluorocarbon rubber at frequencies between 0.1 Hz and 100 Hz, while Figure 10 shows the master curves for the plots of E' and tan d versus frequency generated by shifting the data in Figures 8 and 9 to -5°C. It can be seen in Figure 10 that E' increases rapidly with frequency. For instance in Figure 10, E' increased from 10^7 Pascals (Pa) at 0.1 Hz to $10^{9.5}$ Pa at 1000 Hz at -5°C. This is significant if the fluorocarbon rubber was used to make a boot and operational conditions required service near the freezing point of water and at frequencies greater than 100 Hz.

4.0 Conclusions

5109S neoprene rubber has poor chemical resistance to the three hydrocarbon fill fluids. Therefore it is not recommended that 5109S neoprene rubber be used in applications where it comes in contact with Bayol 35, Voltesso 35, or Marcol 72 fill fluids and retention of mechanical properties and dimensional stability are important.

Fluorel QXA 3770 has good chemical resistance to the fill fluids. However, this elastomer should not be used in applications where the temperature may go below 0°C and at frequencies greater than ~500Hz. Under these conditions the material will begin to become hard and glasslike.

It is recommended that epichlorohydrin rubber be evaluated as a boot rubber. The literature indicates that this rubber has excellent resistance to hydrocarbons and a glass transition temperature well below 0°C.

5.0 References

1. Canadian Centre for Occupational Health and Safety, MSDS Number 248150, Bayol 35, Prepared September 27, 1994.

2. Canadian Centre for Occupational Health and Safety, MSDS Number 243020, Voltesso 35, Prepared October 10, 1995.

3. Canadian Centre for Occupational Health and Safety, MSDS Number 248619, Marcol 72, Prepared September 15, 1994.

4. John A. Hiltz, Richard M. Morchat and Irvin A. Keough, 'A DMTA Study of the Fuel Resistance of Elastomers', Thermochimica Acta, 226 (1993) 143-154.

5. S. B. Harogoppad and T. M. Aminabhavi, Polymer Communications, 32 (1991) 120.

6. S. B. Harogoppad and T. M. Aminabhavi, Polymer, 32 (1991) 870.

7. **The Elements of Polymer Science and Engineering**, A. Rudin, pp 415-417, Academic Press, Toronto, Ontario (1982).
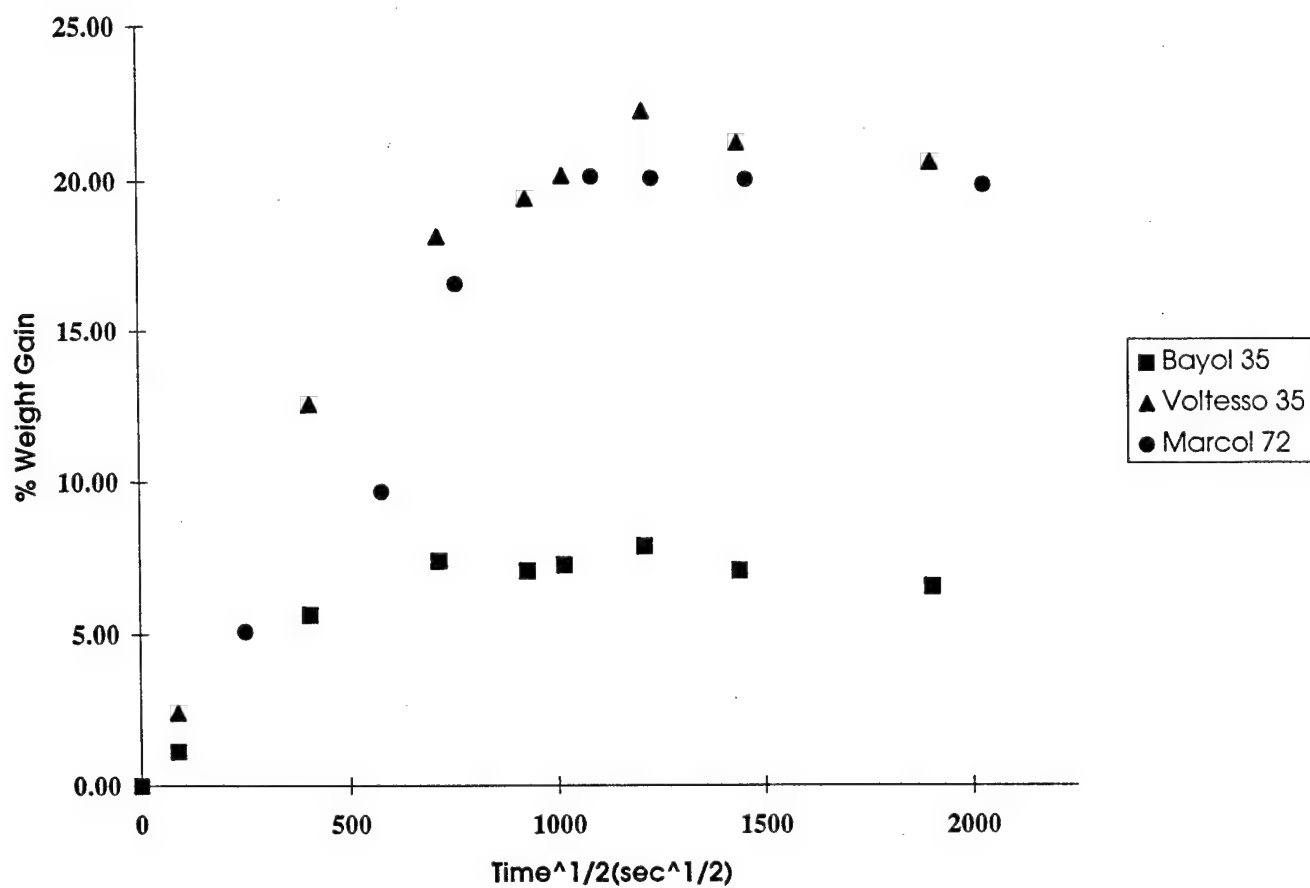
Figure 1 - Plots of percentage weight gain of samples of 5109S neoprene rubber against immersion time$^{1/2}$ in Bayol 35, Voltesso 35, and Marcol 72 fill fluids.
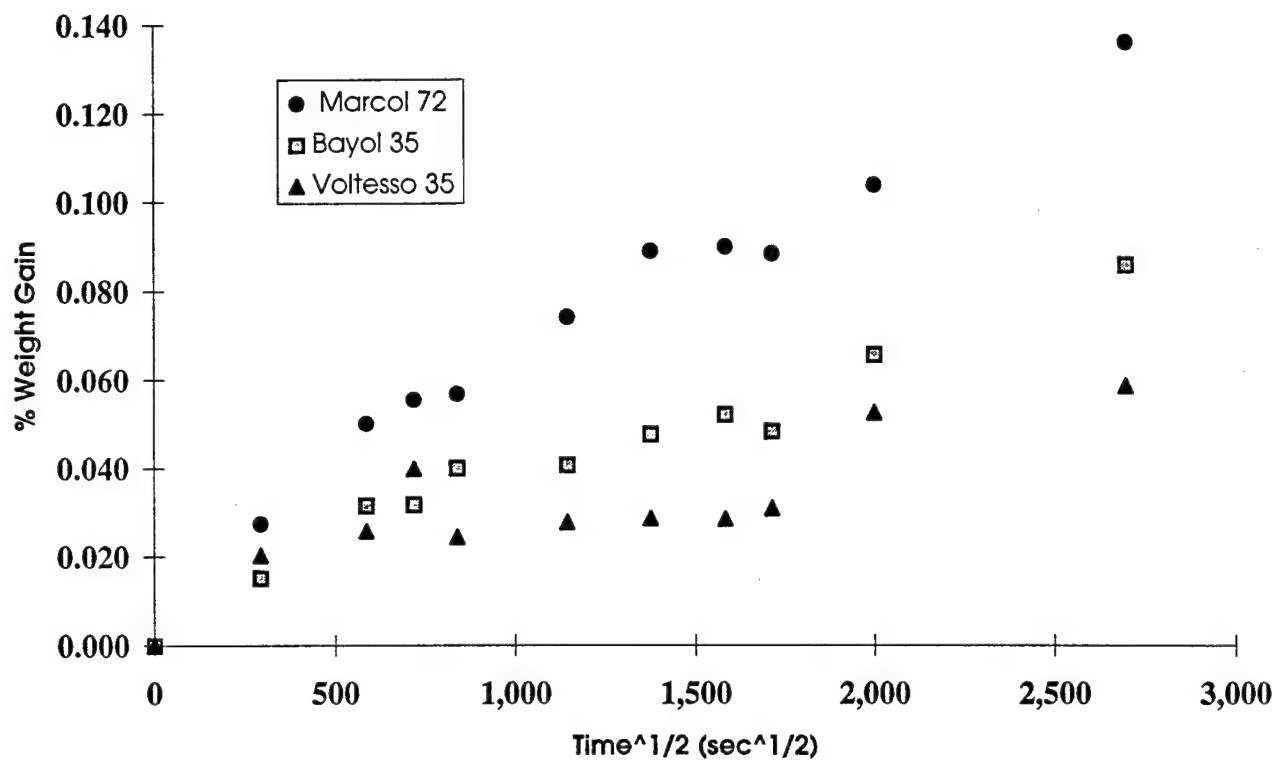
Figure 2 - Plots of percentage weight gain of samples of QXA 3770 fluorocarbon rubber against immersion time$^{1/2}$ in Bayol 35, Voltesso 35, and Marcol 72 fill fluids.
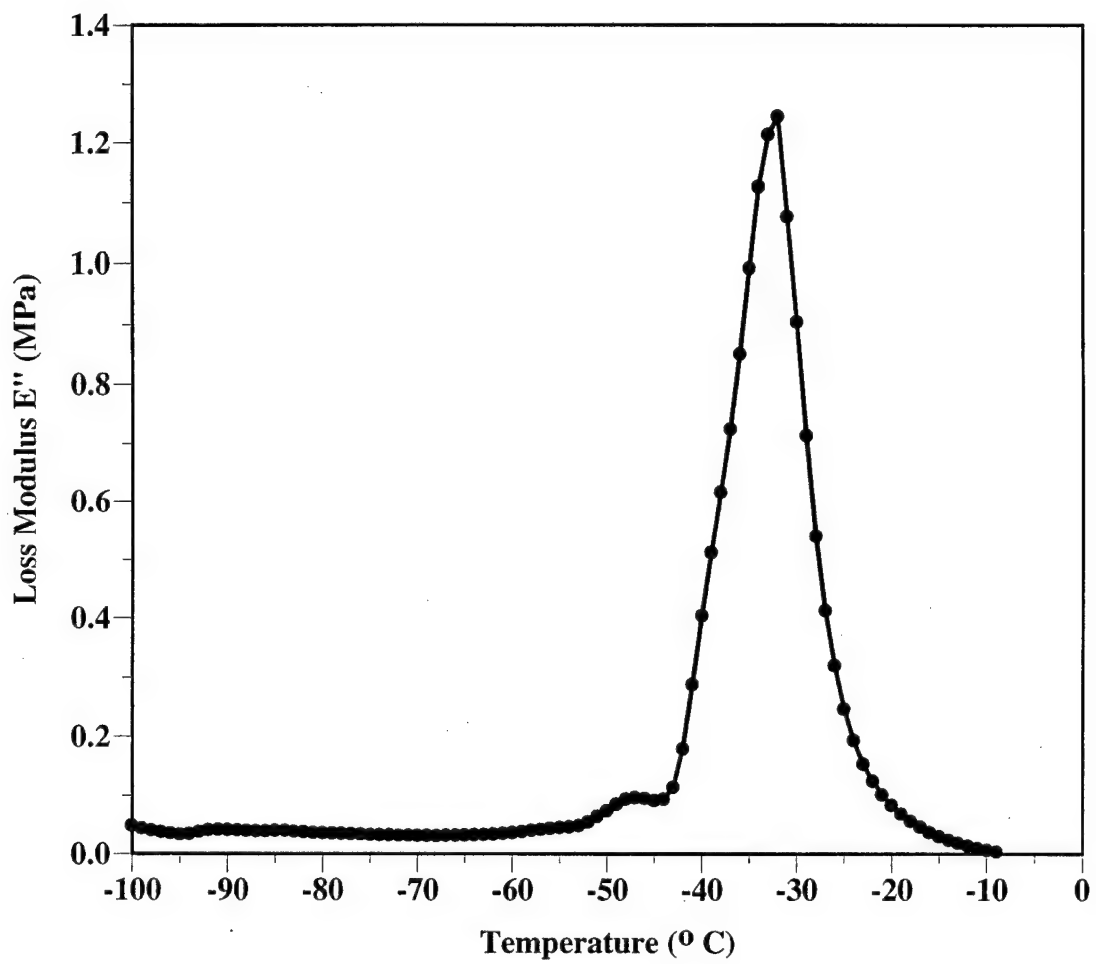
11

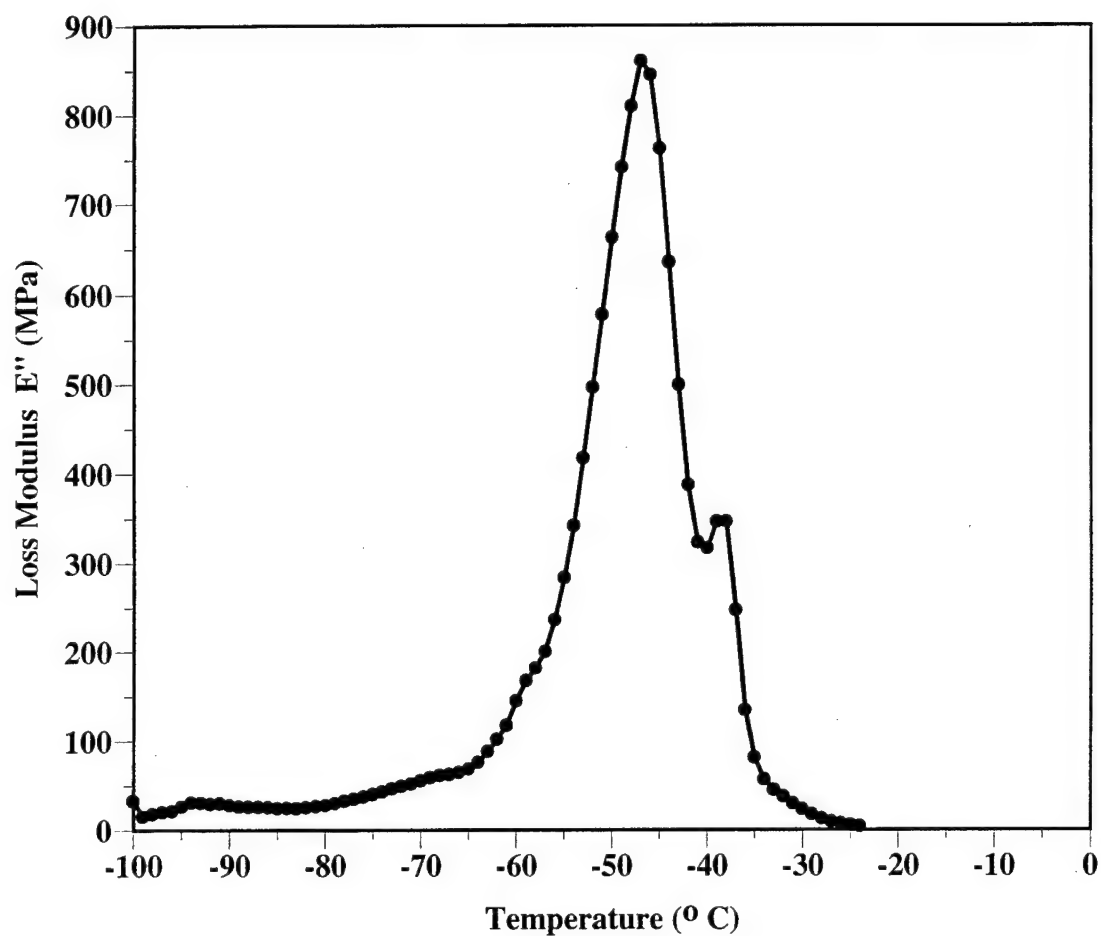Figure 3 - Plot of Loss Modulus (E'') versuses Temperature for 5109S neoprene rubber.

Figure 4 - Plot of Loss Modulus (E") versuses Temperature for 5109S neoprene rubber following immersion in Bayol 35 fill fluid.
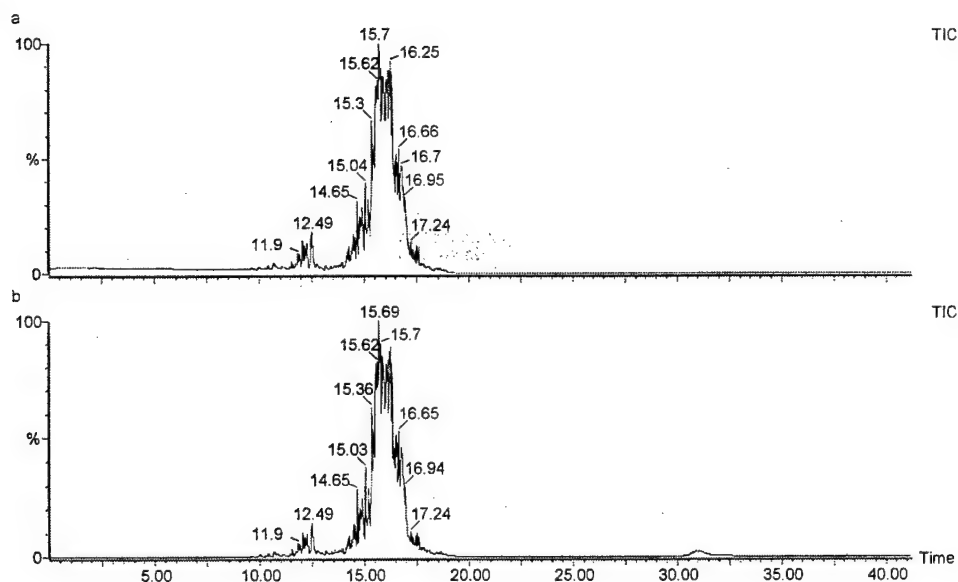
Figure 5 - Chromatograms of Bayol 35 before (a) and after (b) exposure to a Fluorel QXA 3770 fluorocarbon rubber. Note small peak in the chromatogram of the sample exposed to the fluorocarbon rubber at approximately 31.00 minutes.
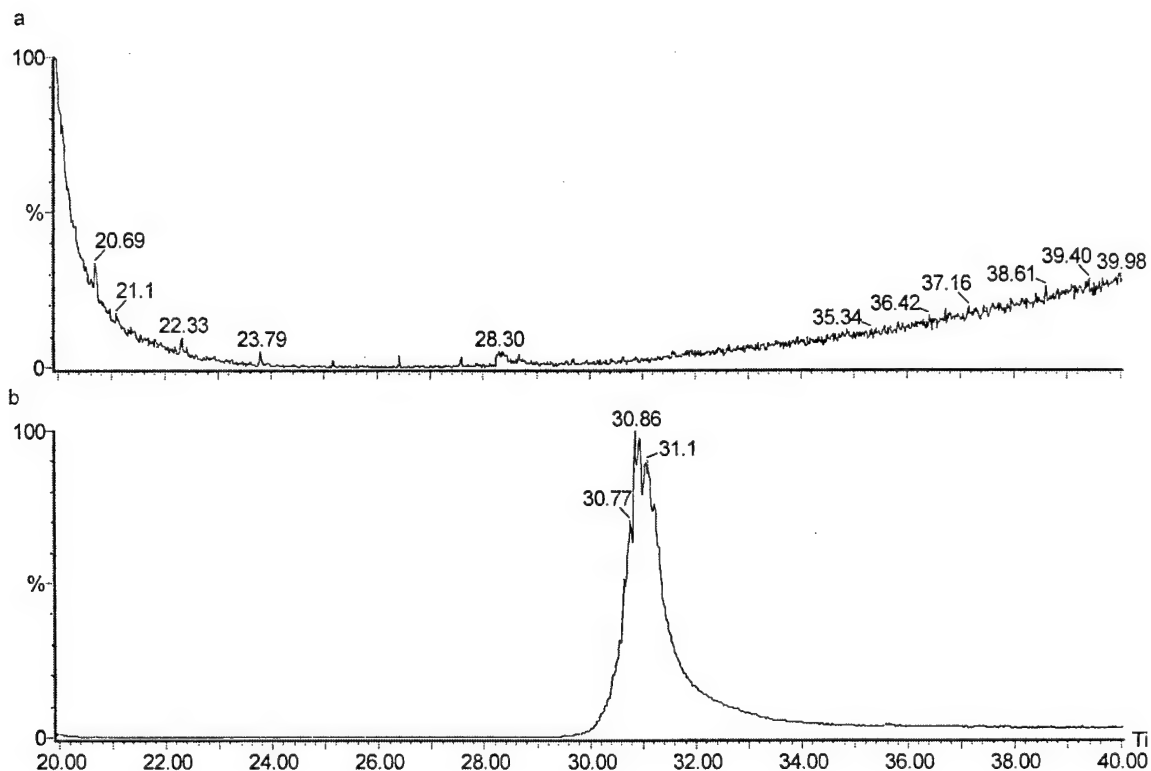

Figure 6 - Expansion of chromatograms (20.00 - 40.00 minutes) of Bayol 35 before (a) and after (b) exposure to Fluorel QXA 3770 fluorocarbon rubber.
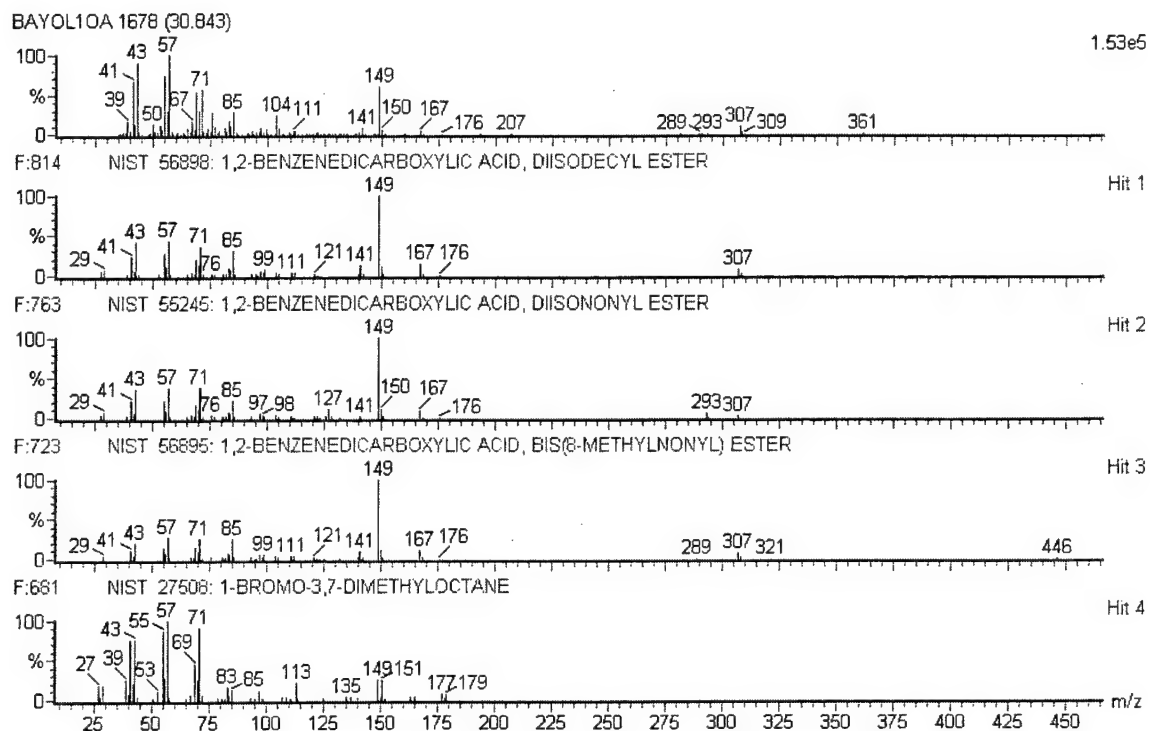
14

Figure 7 - Top trace - Mass spectrum of the compound giving rise to the peak at 30.84 minutes in Figure 8. Bottom traces - Best match mass spectra for the compound.
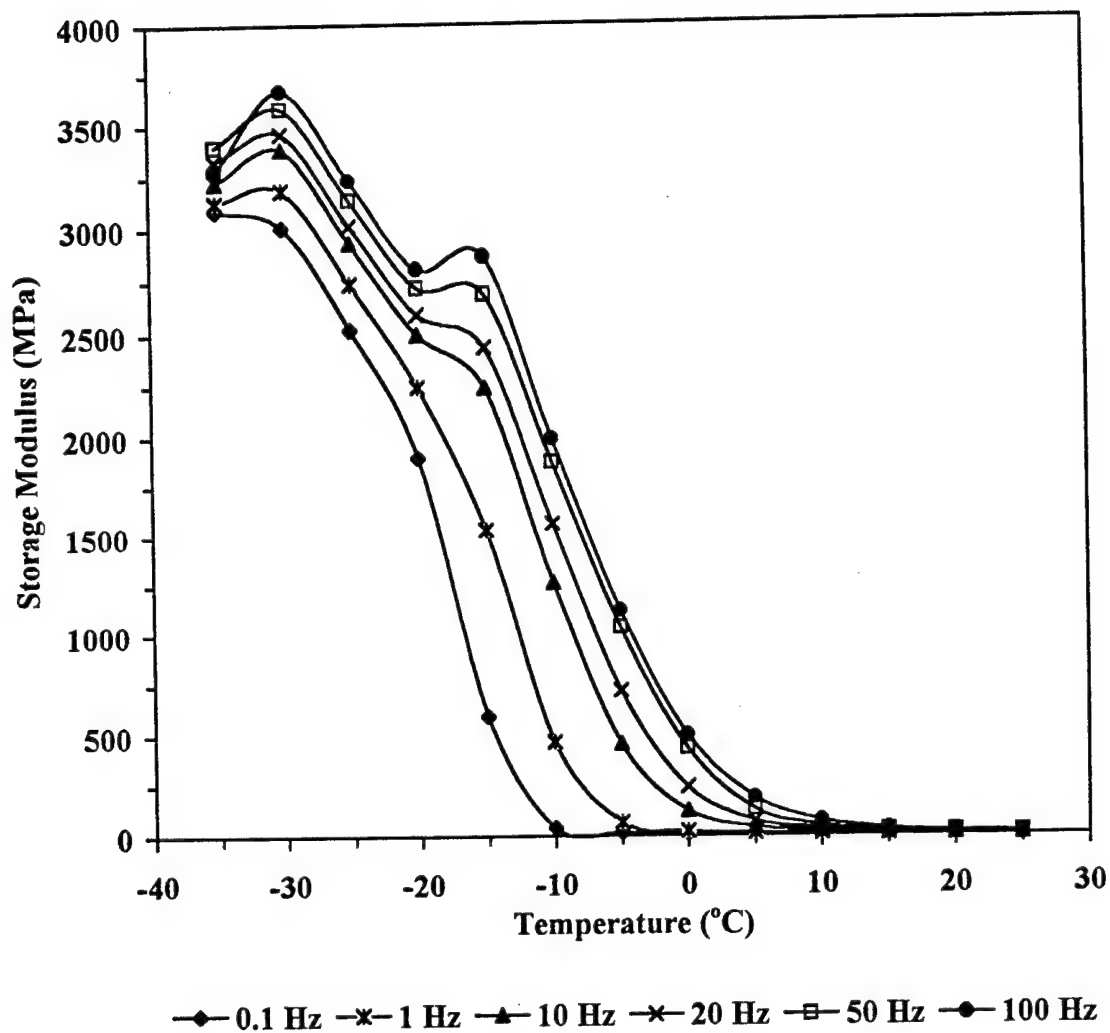
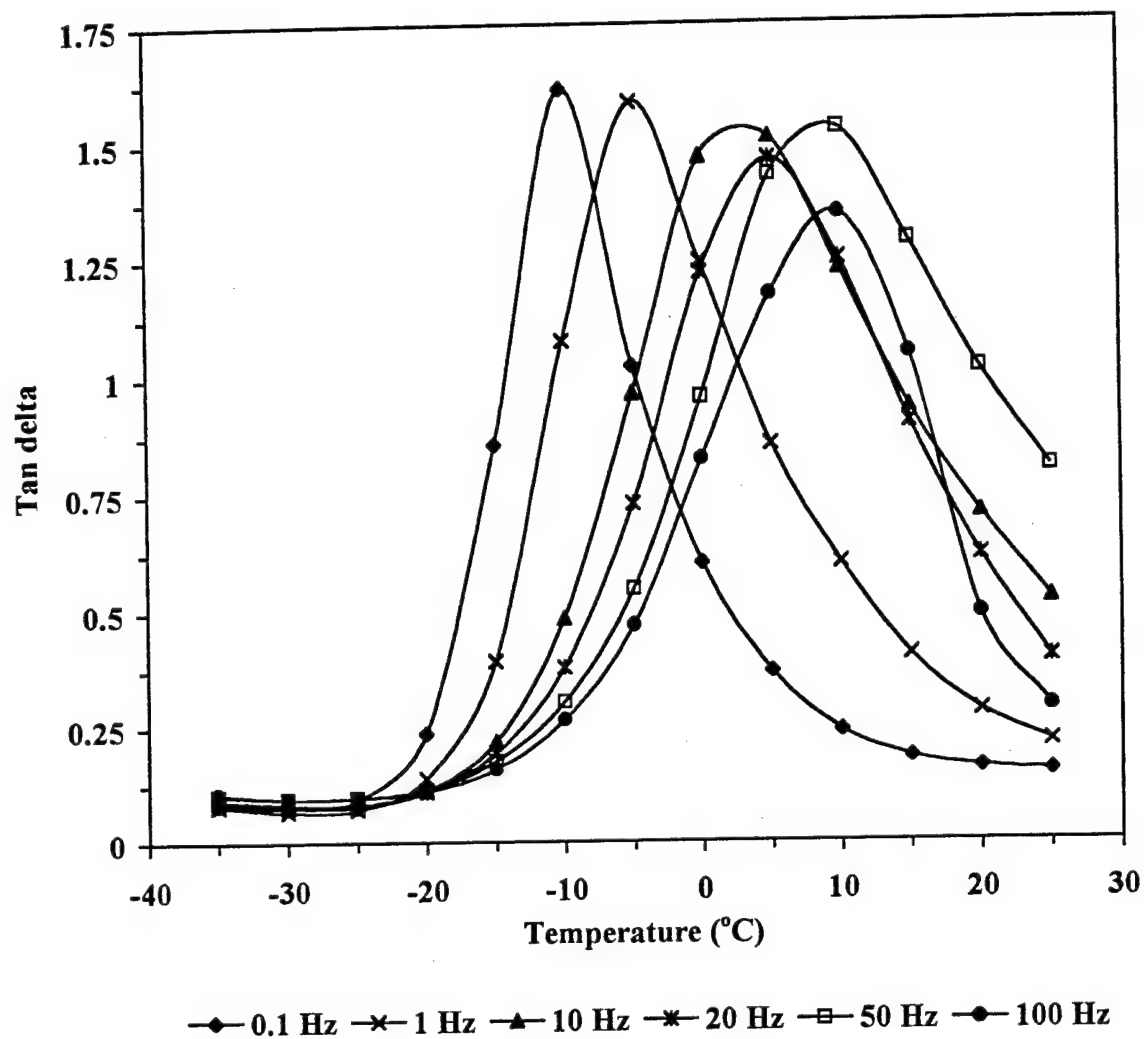Figure 8 - A series of E' versus temperature plots at frequencies between 0.1Hz and 100Hz.

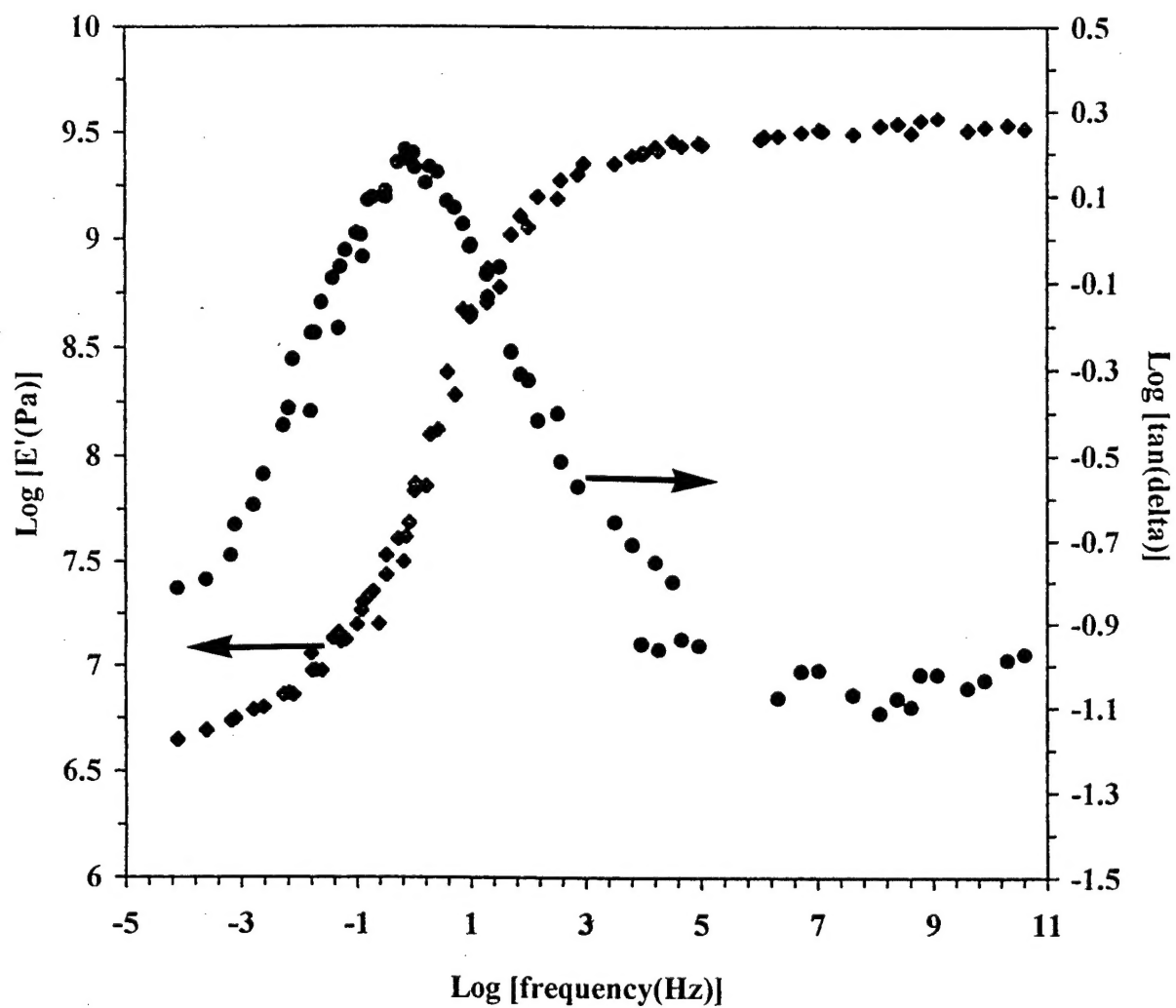Figure 9 - A series of tan δ versus temperature plots at frequencies between 0.1Hz and
100Hz.

Figure 10 - Master curves for E' and tan δ against frequency, QXA 3770 fluorocarbon rubber. Reference temperature is -5° C.

18

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The chemical resistance of two elastomers, 5109S neoprene rubber and QXA 3770 fluorocarbon rubber, to three hydrocarbon-based buoyancy fluids has been assessed by monitoring weight gain and dimensional changes of rubber samples immersed in the fluids. The neoprene rubber absorbed approximately 7%, 20% and 20% by weight Bayol 35, Voltesso 35 and Marcol 72 respectively. The dimensions of the neoprene rubber also increased following immersion in the fluids. The fluorocarbon elastomer absorbed less than 0.15% by weight of the fill fluids, and did not swell following immersion in the fluids. Dynamic mechanical analysis was also used to monitor changes in the properties of the elastomers following immersion. The glass transition temperature (Tg) of the neoprene rubber samples decreased by 15 to 20 degrees following immersion in the fill fluids. Changes in the Tg of the fluorocarbon rubber following immersion were considerably smaller than those observed for the neoprene rubber. The results indicate that the fluorocarbon rubber has good chemical resistance to the fill fluids while the neoprene rubber does not. However, the dynamic properties of the fluorocarbon rubber indicate that it should not be used for low temperature, high frequency applications if rubber-like properties are required.

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Elastomers
neoprene rubber
fluorocarbon rubber
dimensional stability
Dynamic mechanical analysis
fill fluids
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Voltesso 35
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